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4-(2-Carboxyvinyl)pyridinium iodide

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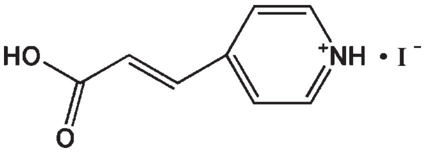
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.028; wR factor = 0.064; data-to-parameter ratio = 19.1.

In the crystal structure of the title salt, $C_8H_8NO_2^+\cdot I^-$, the cations and anions are linked by bifurcated N—H⋯(O,I) hydrogen bonds. A near-linear O—H⋯I hydrogen bond also exists between the cation and anion, resulting in a two-dimensional network. In the cation, the carboxyl group is twisted with respect to the pyridine ring at a dihedral angle of 15.34 (17)°.

Related literature

3-(Pyridin-4-yl)acrylic acid is an intermediate in the synthesis of 3-amino-3-(pyridin-4-yl)propanoic acid, which is of interest as a precursor for the synthesis of novel biologically active compounds, see: Cohen *et al.* (2002); Qu *et al.* (2004).



Experimental

Crystal data

$C_8H_8NO_2^+\cdot I^-$
M_r = 277.05
Monoclinic, $P2_1/n$
a = 4.9685 (10) Å
b = 15.494 (3) Å
c = 12.123 (2) Å
β = 101.48 (3)°
V = 914.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.46 mm^{-1}
T = 293 K
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.492, T_{max} = 0.518
9130 measured reflections
2099 independent reflections
1786 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.064
S = 1.11
2099 reflections
110 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1⋯I1	0.86	3.04	3.652 (3)	130
N1—H1⋯O2^{i}	0.86	2.15	2.819 (3)	134
O1—H1B⋯I1^{ii}	0.82	2.54	3.362 (2)	175

Symmetry codes: (i) $x+\frac{3}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $x-\frac{3}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *PRPKAPPA* (Ferguson, 1999).

This work was supported by Southeast University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2766).

References

Cohen, J. H., Abdel-Magid, A. F., Almond, H. R. Jr & Maryanoff, C. A. (2002). *Tetrahedron Lett.* **43**, 1977–1981.
Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.
Qu, Z.-R., Zhao, H., Wang, Y.-P., Wang, X.-S., Ye, Q., Li, Y.-H., Xiong, R.-G., Abrahams, B. F., Liu, Z.-G. & Xue, Z.-L. (2004). *Chem. Eur. J.* **10**, 54–60.
Rigaku (2005). *CrystalClear.* Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2010). E**66**, o1639 [**doi:10.1107/S1600536810021501**]

4-(2-Carboxyvinyl)pyridinium iodide

D.-Y. Hu

Comment

β-Amino acids are important molecules due to their pharmacological properties. Recently, there has been an increased interest in the enantiomeric preparation of β-amino acids as precursors for the synthesis of novel biologically active compounds (Cohen *et al.*, 2002; Qu *et al.*, 2004). 3-(Pyridin-4-yl)acrylic acid is the intermediate to synthesize 3-amino-3-(pyridin-4-yl)propanoic acid.

The asymmetric unit of the title compound (Fig. 1) contains one 4-(2-carboxyvinyl) pyridinium and one iodate anion. The conformation of the cation is stabilized by an intramolecular N—H···I and C—H···O hydrogen bond (Table 1). In the crystal structure (Fig. 2), molecules are connected by intermolecular N—H···O, O—H···I and C—H···O hydrogen bonds into chains running parallel to the *b* axis (Table 1).

The dielectric constant of the title compound as a function of temperature indicates that the permittivity is basically temperature-independent, suggesting that this compound should be not a real ferroelectrics or there may be no distinct phase transition occurred within the measured temperature range.

Experimental

In a dry, N_2-filled three-necked flask fitted with stirrer, 4-pyridinecarboxaldehyde (1.07 g, 10 mmol) and malonic acid (2.50 g, 24 mmol) were dissolved in pyridine (4 ml) and piperidine (0.1 ml) and this solution was refluxed for 4.5 h and the mixture was then worked up. To the suspension was then added ethylether (5 ml), and the white precipitate was filtered and washed with ethylether (3.5 ml) to give (E)-3-(4-pyridyl)acrylic acid. (E)-3-(4-pyridyl)acrylic acid (0.5 g, 3 mmol) and hydriodic acid (0.43 g, 3 mmol) were dissolved in ethanol (10 ml). After slow evaporation of the solution over a period of 3 days, orange prismatic crystals of the title compound suitable for X-ray diffraction analysis were isolated.

Refinement

All H atoms were placed at calculated positions with C—H = 0.93, N—H = 0.86 and O—H = 0.82 Å, and refined in riding mode with $U_{iso}(H) = 1.5U_{eq}(O)$ and $1.2U_{eq}(C,N)$.

Figures

Fig. 1. A partial packing diagram of the title compound, with the displacement ellipsoids were drawn at the 30% probability level.

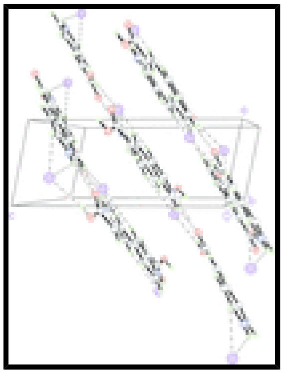


Fig. 2. Packing diagram of the title compound, showing the structure along the *b* axis. Hydrogen bonds are shown as dashed lines.

4-(2-Carboxyvinyl)pyridinium iodide

Crystal data

$C_8H_8NO_2^+\cdot I^-$

M_r = 277.05

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

a = 4.9685 (10) Å

b = 15.494 (3) Å

c = 12.123 (2) Å

β = 101.48 (3)°

V = 914.6 (3) $Å^3$

Z = 4

$F(000)$ = 528.0

D_x = 2.012 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 1866 reflections

θ = 3.2–27.0°

μ = 3.46 mm^{-1}

T = 293 K

Prism, orange

0.20 × 0.20 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer

Radiation source: fine-focus sealed tube

graphite

Detector resolution: 13.6612 pixels mm^{-1}

ω scan

Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)

T_{min} = 0.492, T_{max} = 0.518

9130 measured reflections

2101 independent reflections

1786 reflections with $I > 2\sigma(I)$

R_{int} = 0.046

θ_{max} = 27.5°, θ_{min} = 3.1°

$h = -6\rightarrow6$

$k = -20\rightarrow20$

$l = -15\rightarrow15$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.028

$wR(F^2)$ = 0.064

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$S = 1.11$	$w = 1/[\sigma^2(F_o^2) + (0.0215P)^2 + 0.0886P]$ where $P = (F_o^2 + 2F_c^2)/3$
2099 reflections	$(\Delta/\sigma)_{max} = 0.001$
110 parameters	$\Delta\rho_{max} = 0.54$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.49$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.8838 (5)	0.62959 (16)	0.6895 (2)	0.0397 (6)
H1	1.0091	0.5971	0.7275	0.048*
C8	0.0739 (7)	0.91308 (18)	0.4041 (3)	0.0371 (7)
C3	0.7164 (6)	0.76753 (19)	0.6401 (3)	0.0387 (7)
H3	0.7325	0.8271	0.6486	0.046*
C6	0.2864 (6)	0.78463 (18)	0.4944 (3)	0.0362 (7)
H6	0.1361	0.7556	0.4527	0.043*
C4	0.4734 (6)	0.64341 (19)	0.5602 (3)	0.0396 (8)
H4	0.3229	0.6179	0.5138	0.048*
C5	0.4953 (6)	0.73228 (18)	0.5661 (2)	0.0321 (6)
C2	0.9097 (6)	0.7147 (2)	0.7004 (3)	0.0423 (8)
H2	1.0602	0.7382	0.7492	0.051*
C1	0.6722 (7)	0.59325 (19)	0.6224 (3)	0.0447 (8)
H1A	0.6590	0.5334	0.6176	0.054*
O1	0.1334 (5)	0.99482 (13)	0.3899 (2)	0.0527 (7)
H1B	0.0129	1.0164	0.3417	0.079*
O2	−0.1307 (4)	0.87875 (15)	0.3553 (2)	0.0545 (7)
C9	0.2913 (6)	0.8691 (2)	0.4834 (3)	0.0377 (7)
H9	0.4334	0.9009	0.5262	0.045*
I1	1.17185 (4)	0.413232 (12)	0.682811 (18)	0.04391 (10)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0317 (13)	0.0381 (14)	0.0457 (16)	0.0063 (12)	−0.0011 (12)	0.0076 (12)
C8	0.0352 (17)	0.0361 (17)	0.0362 (18)	0.0011 (13)	−0.0017 (14)	0.0004 (13)

C3	0.0338 (16)	0.0316 (15)	0.0467 (18)	−0.0007 (13)	−0.0019 (13)	−0.0005 (14)
C6	0.0326 (16)	0.0378 (16)	0.0348 (17)	−0.0021 (13)	−0.0019 (13)	−0.0013 (13)
C4	0.0345 (17)	0.0370 (16)	0.0425 (19)	−0.0007 (13)	−0.0042 (14)	−0.0072 (14)
C5	0.0296 (14)	0.0357 (15)	0.0290 (16)	0.0020 (12)	0.0011 (12)	0.0011 (12)
C2	0.0324 (16)	0.0444 (18)	0.044 (2)	−0.0041 (14)	−0.0058 (14)	0.0001 (15)
C1	0.047 (2)	0.0318 (17)	0.052 (2)	−0.0002 (14)	0.0021 (17)	0.0012 (14)
O1	0.0523 (15)	0.0370 (12)	0.0579 (16)	−0.0062 (11)	−0.0152 (12)	0.0125 (11)
O2	0.0441 (14)	0.0390 (12)	0.0663 (17)	−0.0051 (11)	−0.0227 (12)	0.0069 (12)
C9	0.0330 (16)	0.0394 (17)	0.0350 (17)	−0.0017 (13)	−0.0067 (13)	0.0005 (13)
I1	0.04248 (15)	0.03397 (14)	0.04898 (17)	−0.00119 (9)	−0.00609 (11)	−0.00440 (9)

Geometric parameters (Å, °)

N1—C1	1.321 (4)	C6—C5	1.460 (4)
N1—C2	1.330 (4)	C6—H6	0.9300
N1—H1	0.8600	C4—C1	1.360 (4)
C8—O2	1.195 (4)	C4—C5	1.382 (4)
C8—O1	1.319 (3)	C4—H4	0.9300
C8—C9	1.464 (4)	C2—H2	0.9300
C3—C2	1.359 (4)	C1—H1A	0.9300
C3—C5	1.385 (4)	O1—H1B	0.8200
C3—H3	0.9300	C9—H9	0.9300
C6—C9	1.316 (4)		
C1—N1—C2	122.3 (3)	C5—C4—H4	120.0
C1—N1—H1	118.9	C4—C5—C3	118.0 (3)
C2—N1—H1	118.9	C4—C5—C6	118.9 (3)
O2—C8—O1	123.6 (3)	C3—C5—C6	123.0 (3)
O2—C8—C9	124.2 (3)	N1—C2—C3	120.0 (3)
O1—C8—C9	112.2 (3)	N1—C2—H2	120.0
C2—C3—C5	119.7 (3)	C3—C2—H2	120.0
C2—C3—H3	120.1	N1—C1—C4	119.9 (3)
C5—C3—H3	120.1	N1—C1—H1A	120.0
C9—C6—C5	126.0 (3)	C4—C1—H1A	120.0
C9—C6—H6	117.0	C8—O1—H1B	109.5
C5—C6—H6	117.0	C6—C9—C8	120.2 (3)
C1—C4—C5	120.0 (3)	C6—C9—H9	119.9
C1—C4—H4	120.0	C8—C9—H9	119.9

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···I1	0.86	3.04	3.652 (3)	130
N1—H1···O2$^{\mathrm{i}}$	0.86	2.15	2.819 (3)	134
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Symmetry codes: (i) $x+3/2, -y+3/2, z+1/2$; (ii) $x-3/2, -y+3/2, z-1/2$.

Fig. 1

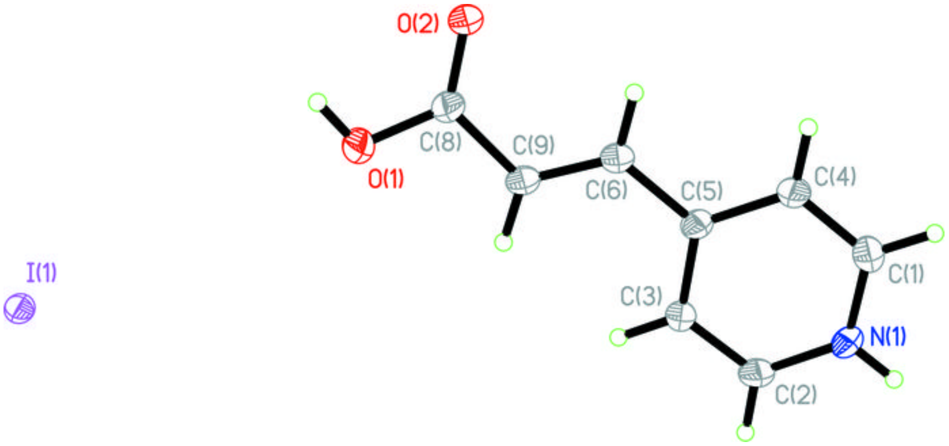

Fig. 2